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EVALUATING THE TENSILE STRENGTH AND K/S VALUE OF COTTON FABRIC DYED WITH NATURAL EXTRACTS OF MADDER ROOT (*RUBIA TINCTORUM)* BY PAD-STEAM PROCESS

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Abstract: Cotton fabric was dyed with natural extracts of madder (*Rubia Tinctorum*) by pad-steam process. To evaluate the effect of different dyeing auxiliaries on the tensile strength and colour properties of cotton fabric, various mordants, UV absorbers, cationizing agents and cross linkers were utilized in the dyeing process. All the auxiliaries were applied both as pre and post padding. Metallic mordants included aluminium sulphate, copper sulphate, ferric chloride, potassium dichromate and hydrated potassium aluminum sulphate or alum.Cationization of cotton with quaternary ammonium compound has improved the tensile strength as well as relative colour strength (K/S) of cotton dyed with this natural dye. Post-treatment of soft polyurethane emulsion managedtoretain the tensile strength of the dyed cotton. Crosslinking agents including methylolation product based on glyoxalmonourein, modified dimethyloldihydroxyethylene urea and modified dihydroxy ethylene urea also made some improvements in the tensile strength of cotton.

Keywords: Tensile strength; madder; K/S value; pad-steam dyeing; cross linkers.

Introduction

Dyeing involves an interaction between a textile, a dye, water and some sort of auxiliary agent. Whether anionic or cationic, dye molecules are applied to textile from aqueous solution. Auxiliaries are the chemicals/compounds which assist the process of dyeing. These may include leveling agents, wetting agents, swelling agents, antifoaming agents, detergents and dispersing agents etc. Dyestuffs and dyeing are as old as textiles themselves. Today, colouration of textiles is a complex and specialized science which involves continuous research and advancement. Until the invention and commercialization of synthetic dyes, the target of textile colouration was mainly achieved by the utilization of natural colours. Synthetic dyes are produced from petrochemical sources through hazardous chemical operations and are supposed to be the cause of many harmful effects on human body such as skin allergies etc. They produce/release toxic and unsafe chemicals during their synthesis which are undesirable. They pose threat of being eco-destructive. In comparison, natural dyes are environment friendly and shades produced by them are soft, uncommon and soothing. Today's consumer has more awareness of

these issues due to the availability of greater number of products with green or organic label (Patel et al, 2002) which has expedited a resurgent interest in utilizing colours obtained from natural sources (Samanta et al, 2009).

The use of various mordants causes different colour hues (H), significant changes in K/S values as well as differences in lightness (L). The strength and stability as well as shade of dye on cotton fabrics largely depend on the choice of mordant selected and the nature of the textile fibre. Ferrous sulphate can provide dark shade when used as mordant with natural dyes. Considering the depth of shade, Ratnapandian et al, (2012) consider post-mordanting procedure as a better option as compared to meta-mordanting or pre- mordanting.

Colour is defined by its attributes of intensity (saturation, chroma), value (brightness) and hue (the name of a colour: red, blue, green, etc.). A computer may require the amount of red, green and blue phosphor emissions to match a colour. CIEL*a*b* and CIEL*C*h^o colour spaces are directly based on the measurement of the parameters or coordinates to specify the colour. The term CIE is the abbreviation from its French title *Commission Internationale de l'Eclairage* which relates to International Commission on Illumination. It has been recognized by ISO for representing the best authority on the subject.

CIEL*a*b* is extensively used in textile industry for colour specification. This colour space has been a general utility for illustrating the relationship among colours since its inception in 1976 (McDonald & Smith, 1995; Ibrahim et al., 2011). The L* axis denote lightness which ranges from 0-100 whereas, a* and b* (which are at right angle to each other) represent the horizontal of the axis. They meet in the centre representing neutral colours (gray, black or white). The axis a* is red at one extreme (positive) and green at the other (negative). Similarly, b* axis has yellow (+b) at one end and blue (-b) at the other. The colour space CIEL*C*h^o contains three axes that is L*, C* and h^o. The axis C* ranges from 0 at the centre of the circle (neutral colours: gray, black or white) to 100 or greater at the edge of the circle (very high chroma or colour purity). Likewise, the axis h^o represents hue. Every possible saturated colour (hue) can be seen around the edge of the circle. The units of h^o are in the form of degrees which range from 0^o (red colour) to 270^o (blue colour).

The ability of a pigment or dye to change the colour of otherwise colourless material is known as colour strength. Its calculations are made on the basis of numerical value related to the amount of light-absorbing material (colourant) contained in a sample (Zarkogianni et al, 2010). This colour value is calculated as the sum of K/S values for the sample. It is proportional to the dye concentration on the fibre. According to the Kubelka–Munk equation:

$$(K/S = (1-R)^2/2R)$$

For all colour matching software, Kubelka-Munk series of equations are mathematical basic. The ratios of total absorbed and scattered light by the dyes are separately measured. Absorption is defined as "K" and scattering is defined as "S". Datacolor SF 600 is equipped with computer added software which provides the computed values of K/S rather than the reflectance etc.

The development in cotton fabric's dyeing properties without altering or degrading the inherent natural tensile strength is of great importance. Whether used for apparel, home furnishing, recreational or industrial purposes, cotton fabric has to experience tensile loads. Mechanical

properties of cotton fabrics such as tensile strength, tear strength and elongation etc. are among the important components in contributing durability and serviceability of the article. Tensile strength is generally expressed in terms of the amount of load or maximum stress which the fabric can withstand before getting ruptured. It is therefore, expressed in the unit of force. In a comparative study on dyeing polyester/cotton fabric, the pad-steam process has been found more effective than the conventional exhaustion process. The pad-steam process has the benefit of less strength loss of the fibers and a reduced amount of water and chemical consumption. This process is therefore, less dangerous to the environment. The dyeability and handle were improved by both processes (Haji et al, 2011).

The effect of different crosslinking agents on natural dyed cotton has been studied by Sheth et al, (2003) with the help of alum, stannous chloride or tartaric acid and dimethyloldihydroxyethyleneurea (DMDHEU) of Clariant Ltd. The colour depth, tear strength and tensile strength of DMDHEU treated fabrics has been found dependable on the type of mordant employed with natural dyes. DMDHEU was applied to Jute dyed fabrics through simultaneous dyeing and finishing (Kamaluddin et al, 2007) to study its physical properties. A direct relationship was found in the dyed fabrics, between the improvement in dye fixation and concentration of resin.

Every fabric undergoes some increase in length under tensile loading while resisting rupture during the stress. The tensile strength of gray fabrics consequently relates to the inherent properties of fibre, yarn count, yarn twist fabric construction and other technological parameters. However, for the final end use, these fabrics need to be treated (dyeing, printing, finishing) for adding the required functional properties. In many cases, various thermal and chemical treatments are carried out during these post-treatments. This can worsen some of the important mechanical properties of fabrics such as strength and elongation.

The present study is aimed to examine the possibility of dyeing cotton fabric with various natural dyes using different dyeing auxiliaries (mordants, crosslinkers, UV absorbers, cationizing agents and some finishing agents). The research work focuses on investigating the colour coordinates and changes in tensile strength of natural dyed cotton fabric in terms of comparison between dye application with and without the above mentioned auxiliaries as well as their pre-dyeing and post-dyeing treatments.

Materials and Method

Extraction of dyes

Solvent extraction procedure of natural colourants was performed with soxhlet apparatus, using ethanol as an organic solvent. For the solvent to evaporate, rotary evaporator was used. The total amount of extracted dye was more than 25 % after weighing the dry extracts. To isolate the colourant from the plants of alkanet, cutch and madder, their roots were chopped manually. The dried and chopped root material of plant was refluxed with 1:1 of water: ethanol (2.5 g in 100ml solution). After 3-4 hours, the suspension was filtered and the filtrate was evaporated. All operations were carried out at room temperature. The extract was suspended in 2 % H_2SO_4 and was refluxed for 2 days. Greenish extracts from alkanet, brown from cutch and reddish-brown

semi solid natural dye extract was obtained from madder.

Methodology of dyeing

Fabric treatment involved various stages including: desizing, scouring and bleaching of fabric to prepare fabric for dyeing; preparation of dyeing formulas; pre-treatment procedure of pad-steam dyeing and post-treatment of pad-steam dyeing procedure.

Preparation of fabric for dyeing

As the fabric was purchased in unfinished (grey) form, it was desized, scoured and bleached. Desizing, which is the process of removing starch or size usually from the warp yarns of woven fabrics, was carried out with desizing agent Bactosol MTN (bacterial amylase, provided by Clariant Ltd). Formulation for desizing involved 2 g/l of desizing agent treated under pH 6-6.5 at 60 °C for 1 hour. Scouring was done to remove impurities which cotton may contain (4-12 % by weight in the form of waxes, ash, proteins and other substances such as reducing sugars etc). For good scouring the proper choices of auxiliaries in alkaline bath is essential. Recipe for scouring comprised 15 g/l of NaOH; 1 g/l of wetting agent, 2g/l of sequestering agent (Polyron by Clariant Ltd) and 1 g/l of detergent. All the ingredients were processed at 80-90 °C for 1 hour. Bleaching was carried out by adding 10 g/l of hydrogen peroxide (H_2O_2) in the formulation. Other substances were: 2 g/l of NaOH (pH 10-10.5), 2 g/l of stabilizer (Pentex GP, by Clariant Ltd., which contains anionic detergent and wetting agent with built- in per oxide stabilizer) and 1 g/l sequestering agent all managed at 80-90 °C for 1 hour. Eventually, the desized, scoured and bleached fabric was cut into small pieces of 10×18 inches each for the application of various dyeing recipes.

Preparation of dyeing liquors

Dye bath for control sample (untreated sample) was prepared by adding 30 g/l of dye together with 1 g/l of wetting agent, Albaflow Conti (penetration accelerant by Huntsman, Textile Effects) and 1 g/l of migration inhibitor, Thermacol MP (by Huntsman, Textile Effects). Dye formulation of pre-mordanting and post- mordanting included 20 g/l of a specific mordant either before or after the padding of dye formula. Likewise, UV absorber, Dicrylan and Fixative finishes were used in the quantity of 30 g/l and crosslinkers in the amount of 50 g/l were also part of pre and post-treatment of dyeing recipes for natural dyes on cotton.

Dyeing with madder natural dye

Dyeing was accomplished through a two bath-two stage, pad steam process or continuous dyeing method which is primarily used for the dyeing of cellulosic fibres such as cotton or viscose rayon as well as their blends. This dyeing process offers some exceptional advantages in the form of simplicity, versatility and flexibility. The process is an economical and efficient way of colouring long runs in particular shades. The application of dye through mordanting process consisted of padding the sample with 20 g/l of every mordant on heavy duty mangle machine by 2 dip-2 nip process. The sample was dried in the air at room temperature and it was subsequently shifted to steamer. Steaming was carried out at 100 °C for 10 minutes. The dried sample was padded with dyeing recipe in the padder by 3 dip-3 nip process. The sample was again dried at room temperature and was taken once

again in a steam box (at 100 oC for 10 minutes). The sequence of the entire operation of pre-padding was as follows (Khattak et al, 2014):

Scheme 1. Sequence of pre-padding dyeing operation.

Scheme 2. Sequence of post-padding dyeing operation.

In the post- treatment process, the sample was padded first with the dye formula. In the second stage, the specific mordant/auxiliary was applied through padding. The same procedure was adopted for the dyeing of cotton with natural dyes using crosslinkers, UV absorbers and other fabric finishes. Auxiliaries such as Albafix WFF and UV-SUN were dried/cured at 30 ^{o}C; all the mordants and Dicrylan at 120 ^{o}C and the crosslinkers were processed in the over feed tenter from 120- 130 ^{o}C for drying or curing.

Machinery used for dyeing

Samples were coloured by continuous dyeing method which required the relevant machinery of pad-steam dyeing. The details of machinery utilized for the applications of various dyeing recipes and fabric testing equipments are as below:

Equipments and standards used for fabric testing

Colour coordinates

Dyed specimens of fabric were examined for relative colour strength and other colour coordinates according to Color iQC & Color iMatch (2012) by computer-aided, Datacolor SF 600 reflectance spectrophotometer with software for colour measurement. Colour

properties were measured as L*, a*, b*, C*, h^{o} and K/S values of the coloured fabric samples.

Dyeing padder

Dye formulations were padded in padding mangles of the thermosol machine.

Heavy duty pad mangle

This machine was used for padding the fabric with various pre and post-treatments (dyeing auxiliaries) required in the dyeing formulas for the present project.

Over feed pin tenter

This machine is designed for heat treatment of a test fabric. It was used to dry the dyed fabric samples according to the standard recommended temperature and time required for each dyeing auxiliaries. It can take 200-450 mm wide test fabric with an adjustable device. Its maximum temperature capacity is 230^{o} C.

Laboratory steamer

The laboratory steamer type DHe is provided with the functionality of (i) steaming with saturated steam (ii) high temperature steaming and (iii) drying, curing or setting. To proceed for the pad-steam dyeing, steaming was predetermined at 100 °C (for 10 minutes) for which the very first function of the steam box was workable.

Tensile strength tester

Tensile strength of cotton fabric samples was determined before and after the application of various dyeing formulas. The procedure was adopted according to the standard method prescribed by American Society for Testing and Materials (ASTM D5035-06, 2006) and Booth, 1996. An electronic tensile strength tester (Testometric 220 D) was used for the purpose. After adjusting the instrument with the required jaws-separation for fabric strength, the strain speed was set to 90 inches per minute.

Results and Discussion

Colour coordinate values of madder dyed cotton

Data in respect of the effect of pre and post-treatments on L*, a*, b*, C*, h^{o} and K/S value of madder dyed cotton samples is enclosed in Table 1.

The darkness of shade (L* value) ranged from 41.30 (control sample) to 56.55 (post-treatment of alum). Post-treatment of aluminium sulphate also gave 52.03 value of L* representing lightness in shade. The values of coordinate, a* were all positive displaying redder shades of dyed samples. The most near towards red was available from the treatment of post alum having a value of 19.92 whereas, the lowest figure of6.05 was observed from the sample which was treated with post ferric chloride. The values of b* from madder dyed cotton were all positive. Chroma or C* was also found the highest (31.12) in the sample dyed with the aid of post-mordanted alum. The same sample also gave better h^{o} (hue) value. However, maximum h^{o} was available from post-mordanted ferric chloride which was 61.64 (Figure 1).

In case of K/S or relative colour strength, maximum value of 8.39 was presented by the same madder dyed cotton sample (post-mordanted ferric chloride) which had a the highest hue (h^{o}) value. The K/S of samples dyed with alum was registered as the lowest among this group of auxiliaries. These were 3.22 and 3.63 for post and pre-treated alum, respectively.

The colour coordinate values of dyed cotton with various finishes and UV absorbers are listed in also listed in Table 1. Lightness was minimum (L* value of 41.23) in the sample dyed with pre-cationizing Albafix. The most light shaded sample turned out to be the one which was post-treated with Rayosan C (79.3 L*). The values of a* from post-treated UV- SUN and both pre and post-treated Rayosan C were negative presenting a greener shade. All other samples posed positive values of a*. The maximum value was attained by control sample showing more redness. The findings from b* were all positive from madder dyed cotton samples of this group displaying yellowness in the tone.

Purity of colour, C* was maximum from the sample dyed with post-treatment of UV-SUN. Hue or shade (h^{o}) ranged from 30.21 (from post-cationizing Albafix WFF) to 92.19 (post-treatment of Rayosan C). The relative colour strength or K/S was greatest from pre-treated UV- SUN sample, followed by pre-treated Albafix (6.67) and untreated sample

(6.62). The L* value from madder dyed cotton with the aid of crosslinkers ranged between 41.30 for control sample to 77.94 for post-treated Fixapret CPF. The values of a* were negatives from the samples post-treated with Fixapret CPF and Knittex RCT presenting greener shade. All other samples got positive a* values, with control sample having the maximum redness (17.02). Similarly, b* was also positive for this group and C* (chroma) was maximum from the sample, post-treated with Knittex RCT. The result of hue or shade was found the highest from the sample post-padded with crosslinker Fixapret CPF. However, the K/S value was found to be the highest from the control sample. The pre-treatment of Knittex RCT produced sample with lowest K/S value among this group.

In the present study, madder exhibited high colour strength values. Bhattacharya and Shah (2000) reported about the use of copper and iron as mordants which exhibited high K/S values. While working on the optimization of colour fastness of henna, Ali et al, (2009) studied the effect of pre and post-mordanting on colour coordinates of dyed fabric. They have mentioned about huge change in hue from both the methods of mordanting. They have also found a great deal of decrease in chroma value in case of iron as mordant. The present study also support the earlier study of Marie et al., (2008) who have worked on the dyeing of cationized cotton with natural dyes and utilized Tanafix SR cationic agent for the enhancement of the dyeability of cotton. They have found increased K/S value of the dyed samples which according to them, kept on increasing with the increase in concentration of the cationic agent.

Tensile strength of cotton dyed with madder root extracts

The data related to the tensile strength of madder dyed cotton is displayed in Figure 2 which reveals that most of the dye formulations resulted in increase in the tensile strength of the samples. Maximum gain in strength due to dyeing was available from the sample dyed with combination of natural extracts of madder roots and Rayosan C, where the increase in strength was 34.7 percent from its pre-treatment and 34.1 percent from its post-treatment.

Table 1 Colour coordinates (properties) of madder dyed cotton using various auxiliaries

Type of auxiliary	Method	Colour properties					
		L*	a*	b*	C*	h°	K/S
$Al_2(SO_4)_3$	Pre-mordanting	48.30	19.64	15.31	29.90	37.94	4.73
	Post-mordanting	52.03	15.77	23.77	28.49	56.40	4.40
$CuSO_4$	Pre-mordanting	46.27	18.87	9.12	20.96	25.80	4.39
	Post-mordanting	43.07	18.85	12.33	22.32	33.19	6.71
$FeCl_3$	Pre-mordanting	45.44	8.35	10.25	13.22	50.84	7.23
	Post-mordanting	43.47	6.05	11.20	12.73	61.64	8.39
$K_2Cr_2O_7$	Pre-mordanting	47.88	16.46	9.72	19.11	30.56	4.15
	Post-mordanting	42.96	16.40	11.00	19.75	33.84	5.75
$KAl(SO_4)_2 \cdot 12H_2O$	Pre-mordanting	51.49	19.92	15.15	25.02	37.26	3.63
	Post-mordanting	59.39	1.95	5.62	5.95	70.84	1.80

Albafix WFF	Pre cationizing	41.23	16.43	11.07	19.81	33.96	6.67
	Post cationizing	42.26	15.80	9.20	18.28	30.21	5.45
Dicrylan	Pre finishing	49.63	15.42	13.33	20.38	40.85	7.71
	Post finishing	71.37	3.09	39.98	40.10	85.58	6.07
UV –SUN	Pre-treatment	47.48	15.93	11.07	19.40	34.79	6.57
	Post-treatment	78.81	-1.09	41.37	41.39	91.22	6.43
Rayosan C	Pre-treatment	78.33	-0.86	39.33	39.84	91.24	4.43
	Post-treatment	79.31	-1.24	40.36	40.39	92.19	4.14
Fixapret CPF	Pre-treatment	47.61	16.08	11.18	19.58	34.81	4.48
	Post-treatment	77.94	-0.63	40.67	40. 67	90.89	4.57
Fixapret F-ECO	Pre-treatment	46.73	16.29	11.92	20.18	36.20	4.95
	Post-treatment	75.15	1.15	39.74	39.76	88.35	4.64
Knittex RCT	Pre-treatment	48.66	15.82	12.17	19.96	37.57	4.35
	Post-treatment	47.61	16.08	11.18	19.58	34.81	4.48
Control sample	Untreated	77.94	-0.63	40.67	40. 67	90.89	4.57

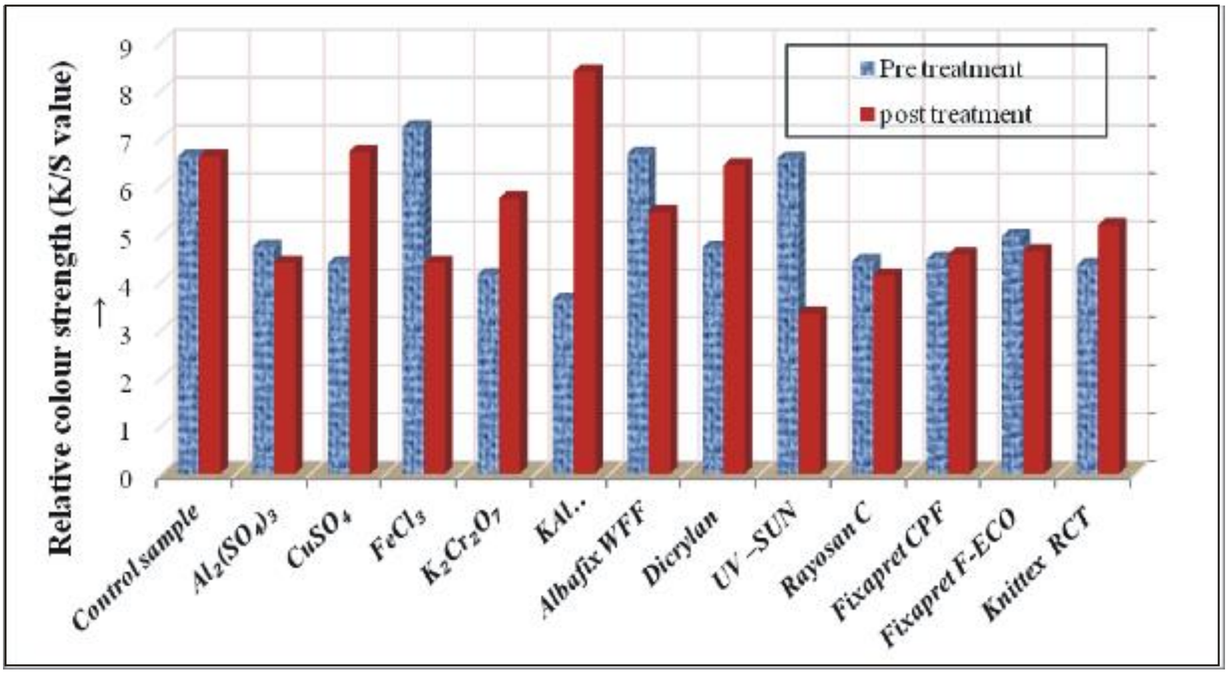

Fig. 1: Effect of various dyeing auxiliaries on the colour strength of cotton samples dyed with root extracts of madder.

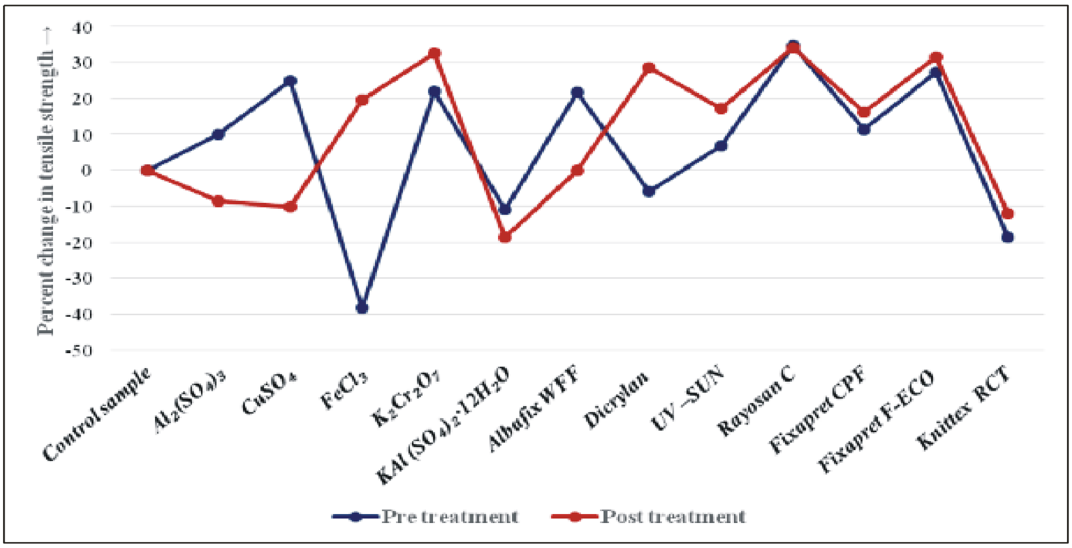

Fig 2: Effect of various dyeing auxiliaries on the tensile strength of cotton samples dyed with root extracts of madder.

Table 2 Paired sample 2- tailed t- test for tensile strength of cotton, dyed with extracts of madder root

Treatments	Paired Differences					t	df	Sig. (2-tailed)
	Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
				Lower	Upper			
*T_1 - T_2	1.42	9.28	2.68	- 7.33	4.47	- 0.53	11	0.61

Table 3 One-Sample t-test: tensile strength of madder dyed cotton.

Treatments	Tensile strength of control sample = 37.5 pounds					
	t	df	Sig. (2-tailed)	Mean Difference	95% Confidence Interval of the Difference	
					Lower	Upper
T_1	1.12	11	0.28	2.63	- 2.52	7.79
T_2	1.91	11	0.08	4.06	- 0.60	8.72

*** T_1 = pre-treatments T_2 = post-treatments**

Increase in tensile strength of 32.5, 31.5, 28.5, 27.2, 24.8 21.9, 21.6, 19.4, 17.0, 16.0 and 11.2 percent from $K_2Cr_2O_7$ (post-padding), Fixapret F-ECO (post- padding), Dicrylan (post- padding), Fixapret F-ECO (pre-treatment), $CuSO_4$ (pre-treatment), $K_2Cr_2O_7$ (pre-treatment), Albafix (pre-treatment), $FeCl_3$ (post- padding), UV –SUN (post- padding) and Fixapret CPF (post-padding), correspondingly. Statistics from t-test shows (Table 2) insignificant result at 5% level of significance between the pre-treated and post-treated samples. Significance testing of the tensile strength of madder dyed cotton fabric samples was also carried out by applying t-test on control sample and pre-treated samples as well between control and post-treated samples (Table 3). The outcome showed insignificant difference between the strength of control and pre-treated samples as well as between the control and post-treated fabric samples. Generally, post padding of auxiliaries had greater positive impact on the tensile strength of cotton dyed with madder root extracts.

Conclusion

Colour properties of madder dyed cotton samples have been enhanced with most of the dyeing auxiliaries, particularly, the post application of alum as mordant. Pre cationization of cotton with quaternary ammonium compound (Albafix WFF) has shown improved colour strength values. Crosslinkers could not bring improvement in this regard. However, a Crosslinking agent Fixapret CPF (methylolation product based on glyoxalmonourein) and Crosslinker Fixapret F ECO (modified dimethyloldihydroxyethylene urea) have tremendously increased the tensile strength of the fabric. Generally, the padding of auxiliaries after dyeing (post-treatments) showed maximum efficiency in appraising the colour strength and tensile strength of the dyed samples.

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